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PATENT ABSTRACTS OF JAPAN

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(71) Applicant: **YOKOGAWA ANALYTICAL SYST
KK**(72) Inventor: **SOGA TOMOYOSHI**(54) **METHOD FOR ANALYZING ANIONIC COMPOUND**

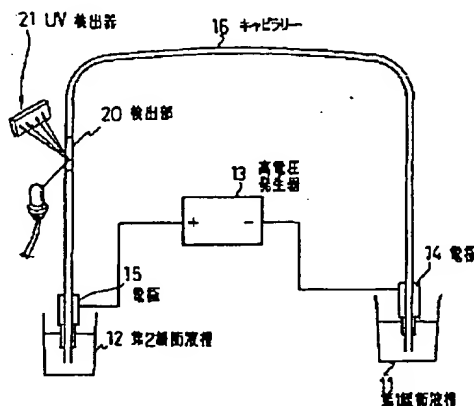
together with the metal, high precision analysis becoming possible.

(57) Abstract:

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PROBLEM TO BE SOLVED: To analyze low concentration anionic compound having a high complex formation ability with metal by using, as a migration buffer liquid, an anionic material having a high complex formation ability to a metal contained in capillary.

SOLUTION: White gold electrodes 14 and 15, cathode side and anode side respectively, of a high voltage generator 13 are immersed in buffer solution baths 11 and 12, respectively. A capillary 16 comprises a molten silica and a polyimide coated layer formed on its outside wall surface, and its start part and end part are submerged in the solution baths 11 and 12, respectively. The coating layer on the liquid bath 12 side of the capillary 16 is peeled off, for forming a detection part 20, and a UV detector is provided. In the solution baths 11 and 12, a buffer liquid having a high complex formation ability to the metal in silica such as 2, 6-pyridine dicarboxylic acid, nitrilotriacetic acid, etc., is held. Thus, when anionic compound sample having a high complex formation ability is analyzed, the buffer liquid and the metal in silica form a complex for masking, thus the sample does not form a complex



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LAIMS

Claim(s)]

Claim 1] Analytical method of the anion nature compound characterized by considering as the anion nature matter which has high complex-forming properties to the metal contained in said capillary tube in said migration buffer solution in the approach of analyzing the anion nature compound which has high complex-forming properties to the metal which fills the migration buffer solution to a capillary tube, impresses high tension to the ends of said capillary tube, and is introduced from one edge of said capillary tube.

Claim 2] Analytical method of the anion nature compound according to claim 1 with which construction material of said capillary tube is characterized by said migration buffer solution being 2,6-pyridine dicarboxylic acid by fuze DOSHIRIKA.

Claim 3] Analytical method of the anion nature compound according to claim 1 with which construction material of said capillary tube is characterized by said migration buffer solution being nitrilotriacetic acid by fuze DOSHIRIKA.

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DETAILED DESCRIPTION

Detailed Description of the Invention]

0001]

[Field of the Invention] This invention fills the migration buffer solution to a capillary tube, impresses high tension to the ends of said capillary tube, and relates to the analytical method which analyzes the anion nature compound which has high complex-forming properties to metals, such as an organic acid introduced from one edge of said capillary tube, and inorganic ion.

0002]

[Description of the Prior Art] Like common knowledge, the capillary electrophoresis which used the capillary tube makes the measuring object the sample of a nano liter (nl) thru/or pico liter (pl) thing ultralow volume, and attracts attention as an analysis means with current and the highest separative power.

0003] Drawing 9 is the block diagram of conventional capillary-electrophoresis equipment. In drawing, the 1st buffer-solution tub in which, as for 1, the migration buffer solution was stored, and 2 are the 2nd buffer-solution tubs in which the migration buffer solution was held.

0004] The dipping of the cathode (cathode) 4 of the high-tension generator 3 is carried out to the 1st buffer-solution tub 1, and the dipping of the anode plate (anode) 5 is carried out to the 2nd buffer-solution tub 2. 6 is capillary tubes, such as for example, fuze DOSHIRIKA, a leader is made into the 1st buffer-solution tub 1, and the dipping of the trailer is carried out to the 2nd buffer-solution tub 2, respectively.

0005] UV on-column detector 7 is formed in the 2nd buffer-solution tub 2 side of a capillary tube 6 as a detector. In such configuration, if the high-tension generator 3 impresses a predetermined electrical potential difference to an anode plate and cathode 5, the migration buffer solution of the 1st buffer-solution tub 1 will move to the 2nd buffer-solution tub 2-way through a capillary tube 6 by electrophoretic force and the electroosmosis style.

0006] Here, if a sample is poured in from the start edge of a capillary tube 6, it will move to the 2nd buffer-solution tub 2-way by electrophoretic force and the electroosmosis style. Passing speed changes with differences in the ionic radius of a sample, or ionicity which hydrated the sample, and a sample becomes band-like and is moved. Consequently, the sample component which moves a capillary tube 6 has the transit time according to the hydrated difference in the ionic radius of a sample, or ionicity, and detects this with UV on-column detector 7.

0007]

[Problem(s) to be Solved by the Invention] In the capillary-electrophoresis equipment of the above-mentioned configuration, since it migrated an electroosmosis style (henceforth EOF), and reversely as shown in drawing 10 (a), in the case of the anion nature compound in which a sample does not have strong UV absorptivity like an organic acid or an inorganic anion, analysis time amount was not able to be taken, or it was not able to be detected.

0008] So, in JP,6-94677,A, the 4th class amines, such as a cetyl trimethylammonium salt's picture, are added to the migration buffer solutions, such as a phthalic acid, trimellitic acid, and a chromic acid, and the method of reversing EOF. is shown in drawing 10 (b) is indicated.

0009] In such an approach, for UV absorption of the migration buffer solution, although a background is fixed, if a detected material without strong UV absorption passes, an absorbance will become low and it will serve as a negative peak. The signal of this negative peak is reversed, and it is made a forward peak and detects.

0010] By the way, generally the impurity is contained in the capillary tube 6 of fuze DOSHIRIKA. Drawing 11 is drawing showing the metal ion concentration in the fuze DOSHIRIKA capillary tube 6 measured with inductively-

coupled-plasma-source-mass-spectrometry equipment (ICP-MS). This drawing shows that metals, such as iron (Fe), and aluminum (aluminum), Na (sodium), Cr (chromium), Mg (magnesium), are contained in the fuze DOSHIRIKA capillary tube 6 as an impurity.

0011] A phthalic acid, above-mentioned trimellitic acid, the above-mentioned chromic acid, etc. are used as the migration buffer solution here. If in the case of the anion nature compound with which a sample has high complex-forming properties, such as an organic acid (for example, oxalic acid, a citric acid, a malic acid, and ethylenediaminetetraacetic acid) and an inorganic anion, sample concentration becomes about 50 ppm or less whenever the above-mentioned Metals contained in fuze DOSHIRIKA of a capillary tube 6 as an impurity, such as iron and aluminum, and these anion nature compound form a complex, and there is a trouble that analysis of a sample cannot be performed.

0012] This invention was made in view of the above-mentioned trouble, and the object has a metal and complex-forming properties in offering the analytical method and the analysis apparatus of a strong low concentration anion nature compound.

0013]

Means for Solving the Problem] Said technical problem is solvable with the following invention.

1) In the approach of analyzing the metal which filled the migration buffer solution to the 1st invention capillary tube, compressed high tension to the ends of said capillary tube, and was introduced from one edge of said capillary tube, and the anion nature compound which has high complex-forming properties, it is the analytical method of the anion nature compound used as the anion nature matter which has high complex-forming properties to the metal contained in said capillary tube in said migration buffer solution.

0014] Since the migration buffer solution is the anion nature matter which has high complex-forming properties to the metal contained in said capillary tube, it will form a complex strongly with the metal contained in the capillary tube, and will mask these metals.

0015] Therefore, even if the sample has a metal and high complex-forming properties, a sample does not form a metal and a complex, but even if a sample is low concentration, high analysis of precision is attained.

2) In the 2nd invention invention of the 1st, it is the analytical method of the anion nature compound said whose migration buffer solution the construction material of said capillary tube is fuze DOSHIRIKA, and is 2,6-pyridine dicarboxylic acid.

0016] There are iron, aluminum, etc. as a metal contained in fuze DOSHIRIKA used for a capillary tube. On the other hand, 2,6-pyridine dicarboxylic acid has high ** stable frequency to these iron, aluminum, etc., and these metals will be masked by using for the migration buffer solution.

0017] Therefore, even if the sample has a metal and high complex-forming properties, a sample does not form a metal and a complex, but even if a sample is low concentration, high analysis of precision is attained.

3) In the 3rd invention invention of the 1st, it is the analytical method of the anion nature compound said whose migration buffer solution the construction material of said capillary tube is fuze DOSHIRIKA, and is nitrilotriacetic acid.

0018] There are iron, aluminum, etc. as a metal contained in the fuze DOSHIRIKA capillary tube. On the other hand, nitrilotriacetic acid has high ** stable frequency to these iron, aluminum, etc., and these metals will be masked by using for the migration buffer solution.

0019] Therefore, even if the sample has a metal and high complex-forming properties, a sample does not form a metal and a complex, but even if a sample is low concentration, high analysis of precision is attained.

0020]

Embodiment of the Invention] Next, one example of this invention is explained using a drawing. It is the sectional view of a capillary tube [in / drawing 1 , and / in drawing 2 / drawing 1]. [the block diagram of the electrophoresis apparatus of one example of this invention]

0021] In drawing 1 , the 1st buffer-solution tub in which, as for 11, the migration buffer solution was stored, and 12 are the 2nd buffer-solution tubs in which the migration buffer solution was stored. The dipping of the platinum electrode 14 by the side of the cathode of the high-tension generator 13 is carried out to the 1st buffer-solution tub 11, and the dipping of the platinum electrode 15 by the side of an anode plate is carried out to the 2nd buffer-solution tub 12.

0022] 16 is a capillary tube, a leader is made into the 1st buffer-solution tub 11, and the dipping of the trailer is carried out to the 2nd buffer-solution tub 12, respectively. The capillary tube 16 of this example consists of fuze DOSHIRIKA 17 and a polyimide enveloping layer 18 formed in the external wall surface of fuze DOSHIRIKA 17.

0023] An enveloping layer 18 exfoliates and the detecting element 20 is formed by the 2nd buffer-solution tub 12 side of

capillary tube 16. UV detector 21 which consists of a UV and a diode array detector is formed in this detecting element 0.

0024] The migration buffer solutions, such as 2,6-pyridine dicarboxylic acid and nitrilotriacetic acid, are stored by the 1st and 2nd buffer-solution tubs 11 and 12. Drawing 3 is drawing showing the complex stability constant to the metal of the compound used for an organic acid or the migration buffer solution. For example, it turns out that 2,6-pyridine dicarboxylic acid has high complexing to the iron contained in fuze DOSHIRIKA 17, aluminum, sodium, and magnesium.

0025] Therefore, the 2,6-pyridine dicarboxylic acid which is the migration buffer solution when analyzing the sample of the anion nature compound which has high complex-forming properties, such as an organic acid (for example, oxalic acid, a citric acid, a malic acid, and ethylenediaminetetraacetic acid) and an inorganic anion, The metal (iron, aluminum, sodium, magnesium) contained in the capillary tube 16 forms a complex, the metal contained in the capillary tube 16 is masked, a metal and a complex are not formed, but even if a sample is low concentration, the high analysis of precision of sample is attained.

0026]

Example] The detail specification of the equipment of a configuration as shown in drawing 1 is described below.

1) Capillary tube 16 bore of 75 micrometers, the outer diameter of 350 micrometers, the overall length of 80.5cm, 72cm lie length from the 1st buffer-solution tub 11 to a detecting element 20) (2) of effective length Buffer-solution 5mM 2,6-pyridine dicarboxylic acid, 0.5mM(s) Cetyl trimethylammonium star's picture pH 5.6 (3) Applied voltage of -25kV (4) (5) which pours the preconditioning migration buffer solution for 4 minutes Temperature of 20 degrees C (6) The impregnation approach inflatable flexible bag technique of a sample is used. 50mbars -- impregnation during 2 seconds 7) the UV detector 21 measurement wavelength of 200nm, and 350nm of reference wave length -- an indirect extinction method -- detection [experiment 1] -- the result of having analyzed the organic-acid standard solution which has the following organic-acid anions is shown in drawing 4.

0027] 1. Chloride Ion (Chloride) 2. Sulfate ion (sulfate) 3. Oxalate ion (oxalate) 4. Formic-acid ion (formate) 5. Malic-acid ion (malate) 6. Citric-acid ion (citrate) 7. Succinic-acid ion (succinate) 8. Pyruvic-acid ion (pyruvate) 9. Acetic-acid ion (acetate) 10. Lactic-acid ion (lactate) 11. Phosphoric-acid ion (phosphate) 12. As shown in pyroglutamic acid (pyroglutamate) drawing 5 When the 2,6-pyridine-dicarboxylic-acid buffer solution was used, the repeatability (relative standard deviation) of transit time was less than 0.13%, and the repeatability (relative standard deviation) of area (peak area) was also dramatically as good as 0.6 - 2.6%.

The organic acid in Biel diluted 5 times with water analyzes using 2,6-pyridine dicarboxylic acid as the [experiment 2] buffer solution, and a result is shown in drawing 6. Analysis of iron, aluminum, etc. the oxalate ion that is easy to form a complex, citric-acid ion, etc. was attained.

Experiment 3] The inorganic anion liquid of the following presentations is analyzed and a result is shown in drawing 7.

0028] 1. Chloride Ion (Cl-) 2. Sulfate Ion (SO42-) 3. Chloric-Acid Ion (ClO3-) 4. Bromate (BrO3-) 5. Chlorous-Acid Ion (ClO2-) 6. Hypophosphorous Acid Ion (PO23-) 7. Iodic-Acid Ion (IO3-) 8. phosphorous acid ion (PO33-) 9. phosphoric-acid ion (PO43-) -- by this approach, it became possible to measure these inorganic anions.

As the [experiment 4] migration buffer solution, using 2,6-pyridine dicarboxylic acid (PDC), and the phthalic acid and trimellitic acid which are used conventionally well to an organic acid, the organic-acid standard solution which used these comparisons in the experiment 1 performs, and a result is shown in drawing 8 (a) - (b).

0029] If 2,6-pyridine dicarboxylic acid is used for the migration buffer solution, as the experiment 1 explained, all organic-acid ion will be detected. On the other hand, when a phthalic acid and trimellitic acid were used for the migration buffer solution, by 50 or less mg/l, neither the oxalate ion of peak No.3 nor the citric-acid ion of No.6 was detected for the concentration of an organic acid. Furthermore, the peak of the malic-acid ion of No.5 also became small, and the effectiveness of 2,6-pyridine dicarboxylic acid was checked.

0030]

Effect of the Invention] As explained in detail above, according to the analytical method of the anion nature compound of the 1st invention, by considering as the anion nature matter which has high complex-forming properties to the metal contained in a capillary tube in the migration buffer solution, the migration buffer solution will form a complex strongly with the metal contained in the capillary tube, and will mask these metals.

0031] Therefore, even if the sample has a metal and high complex-forming properties, a sample does not form a metal and a complex, but even if a sample is low concentration, high analysis of precision is attained. According to the

analytical method of the anion nature compound of the 2nd invention, in the 1st invention, said migration buffer solution considered [the construction material of said capillary tube] as 2,6-pyridine dicarboxylic acid by fuze DOSHIRIKA. There are iron, aluminum, etc. as a metal contained in fuze DOSHIRIKA used for a capillary tube. On the other hand, 2,6-pyridine dicarboxylic acid has high ** stable frequency to these iron, aluminum, etc., and will mask these metals. 0032] Therefore, even if the sample has a metal and high complex-forming properties, a sample does not form a metal and a complex, but even if a sample is low concentration, high analysis of precision is attained. According to the analytical method of the anion nature compound of the 3rd invention, in the 1st invention, said migration buffer solution considered [the construction material of said capillary tube] as nitrilotriacetic acid by fuze DOSHIRIKA. 0033] There are iron, aluminum, etc. as a metal contained in the fuze DOSHIRIKA capillary tube. On the other hand, nitrilotriacetic acid has high ** stable frequency to these iron, aluminum, etc., and will mask it. 0034] Therefore, even if the sample has a metal and high complex-forming properties, a sample does not form a metal and a complex, but even if a sample is low concentration, high analysis of precision is attained.

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TECHNICAL FIELD

Field of the Invention] This invention fills the migration buffer solution to a capillary tube, impresses the high voltage to the both ends of said capillary tube, and relates to the analytical method which analyzes the anion nature compound which has high complex-forming properties to metals, such as an organic acid introduced from one edge of said capillary tube, and inorganic ion.

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PRIOR ART

Description of the Prior Art] Like common knowledge, the capillary electrophoresis which used the capillary tube makes measuring object the sample of a nano liter (nl) thru/or pico liter (pl) thing ultralow volume, and attracts attention as an analysis means with current and the highest separative power.

0003] Drawing 9 is the block diagram of conventional capillary-electrophoresis equipment. In drawing, the 1st buffer-solution tub in which, as for 1, the migration buffer solution was stored, and 2 are the 2nd buffer-solution tubs in which the migration buffer solution was held.

0004] The dipping of the cathode (cathode) 4 of the high-voltage generator 3 is carried out to the 1st buffer-solution tub, and the dipping of the anode plate (anode) 5 is carried out to the 2nd buffer-solution tub 2. 6 is capillary tubes, such as for example, fused silica, a leader is made into the 1st buffer-solution tub 1, and the dipping of the trailer is carried out to the 2nd buffer-solution tub 2, respectively.

0005] UV on-column detector 7 is formed in the 2nd buffer-solution tub 2 side of a capillary tube 6 as a detector. In such configuration, if the high-voltage generator 3 impresses a predetermined electrical potential difference to an anode plate and cathode 5, the migration buffer solution of the 1st buffer-solution tub 1 will move to the 2nd buffer-solution tub 2-way through a capillary tube 6 by electrophoretic force and the electroosmosis style.

0006] Here, if a sample is poured in from the start edge of a capillary tube 6, it will move to the 2nd buffer-solution tub 2-way by electrophoretic force and the electroosmosis style. Passing speed changes with the ionic radii of a sample and the differences in ionicity which hydrated the sample, and a sample becomes band-like and is moved. Consequently, the sample component which moves a capillary tube 6 has the transit time according to the ionic radius of a sample and the difference in ionicity which were hydrated, and detects this with UV on-column detector 7.

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EFFECT OF THE INVENTION

Effect of the Invention] As explained in detail above, according to the analytical method of the anion nature compound of the 1st invention, by considering as the anion nature matter which has high complex-forming properties to the metal contained in a capillary tube in the migration buffer solution, the migration buffer solution will form a complex strongly with the metal contained in the capillary tube, and will mask these metals.

0031] Therefore, even if the sample has a metal and high complex-forming properties, a sample does not form a metal and a complex, but even if a sample is low concentration, high analysis of precision is attained. According to the analytical method of the anion nature compound of the 2nd invention, in the 1st invention, said migration buffer solution considered [the quality of the material of said capillary tube] as 2,6-pyridine dicarboxylic acid by fuze DOSHIRIKA.

There are iron, aluminum, etc. as a metal contained in fuze DOSHIRIKA used for a capillary tube. On the other hand, 2,6-pyridine dicarboxylic acid has high ** stable frequency to these iron, aluminum, etc., and will mask these metals.

0032] Therefore, even if the sample has a metal and high complex-forming properties, a sample does not form a metal and a complex, but even if a sample is low concentration, high analysis of precision is attained. According to the analytical method of the anion nature compound of the 3rd invention, in the 1st invention, said migration buffer solution considered [the quality of the material of said capillary tube] as nitrilotriacetic acid by fuze DOSHIRIKA.

0033] There are iron, aluminum, etc. as a metal contained in the fuze DOSHIRIKA capillary tube. On the other hand, nitrilotriacetic acid has high ** stable frequency to these iron, aluminum, etc., and will mask it.

0034] Therefore, even if the sample has a metal and high complex-forming properties, a sample does not form a metal and a complex, but even if a sample is low concentration, high analysis of precision is attained.

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TECHNICAL PROBLEM

Problem(s) to be Solved by the Invention] In the capillary-electrophoresis equipment of the above-mentioned configuration, since it migrated contrary to the electroendosmose style (henceforth EOF) as shown in drawing 10 (a), in the case of the anion nature compound in which a sample does not have strong UV absorptivity like an organic acid or an inorganic anion, analysis time amount was not able to be taken, or it was not able to be detected.

0008] So, in JP,6-94677,A, the 4th class amines, such as a cetyl trimethylammonium star's picture, are added to the migration buffer solutions, such as a phthalic acid, trimellitic acid, and a chromic acid, and the method of reversing EOF, as shown in drawing 10 (b) is indicated.

0009] In such an approach, for UV absorption of the migration buffer solution, although a background is fixed, if a detected material without strong UV absorption passes, an absorbance will become low and it will serve as a negative peak. The signal of this negative peak is reversed, and it is made a forward peak and detects.

0010] By the way, generally the impurity is contained in the capillary tube 6 of fuze DOSHIRIKA. Drawing 11 is drawing showing the metal ion concentration in the fuze DOSHIRIKA capillary tube 6 measured with inductively-coupled-plasma-source-mass-spectrometry equipment (ICP-MS). This drawing shows that metals, such as iron (Fe), and aluminum (aluminum), Na (sodium), Cr (chromium), Mg (magnesium), are contained in the fuze DOSHIRIKA capillary tube 6 as an impurity.

0011] A phthalic acid, above-mentioned trimellitic acid, the above-mentioned chromic acid, etc. are used as the migration buffer solution here. If in the case of the anion nature compound with which a sample has high complex-forming properties, such as an organic acid (for example, oxalic acid, a citric acid, a malic acid, and ethylenediaminetetraacetic acid) and an inorganic anion, sample concentration becomes about 50 ppm or less whenever noble metals contained in fuze DOSHIRIKA of a capillary tube 6 as an impurity, such as iron and aluminum, and these anion nature compound form a complex, and there is a trouble that analysis of a sample cannot be performed.

0012] This invention was made in view of the above-mentioned trouble, and the purpose has a metal and complex-forming properties in offering the analytical method and the analysis apparatus of a strong low concentration anion nature compound.

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MEANS

Means for Solving the Problem] Said technical problem is solvable with the following invention.

1) In the approach of analyzing the metal which filled the migration buffer solution to the 1st invention capillary tube, impressed the high voltage to the both ends of said capillary tube, and was introduced from one edge of said capillary tube, and the anion nature compound which has high complex-forming properties, it is the analytical method of the anion nature compound used as the anion nature matter which has high complex-forming properties to the metal contained in said capillary tube in said migration buffer solution.

0014] Since the migration buffer solution is the anion nature matter which has high complex-forming properties to the metal contained in said capillary tube, it will form a complex strongly with the metal contained in the capillary tube, and will mask these metals.

0015] Therefore, even if the sample has a metal and high complex-forming properties, a sample does not form a metal and a complex, but even if a sample is low concentration, high analysis of precision is attained.

2) In the 2nd invention invention of the 1st, it is the analytical method of the anion nature compound said whose migration buffer solution the quality of the material of said capillary tube is fuze DOSHIRIKA, and is 2,6-pyridine dicarboxylic acid.

0016] There are iron, aluminum, etc. as a metal contained in fuze DOSHIRIKA used for a capillary tube. On the other hand, 2,6-pyridine dicarboxylic acid has high ** stable frequency to these iron, aluminum, etc., and these metals will be masked by using for the migration buffer solution.

0017] Therefore, even if the sample has a metal and high complex-forming properties, a sample does not form a metal and a complex, but even if a sample is low concentration, high analysis of precision is attained.

3) In the 3rd invention invention of the 1st, it is the analytical method of the anion nature compound said whose migration buffer solution the quality of the material of said capillary tube is fuze DOSHIRIKA, and is nitrilotriacetic acid.

0018] There are iron, aluminum, etc. as a metal contained in the fuze DOSHIRIKA capillary tube. On the other hand, nitrilotriacetic acid has high ** stable frequency to these iron, aluminum, etc., and these metals will be masked by using for the migration buffer solution.

0019] Therefore, even if the sample has a metal and high complex-forming properties, a sample does not form a metal and a complex, but even if a sample is low concentration, high analysis of precision is attained.

0020]

Embodiment of the Invention] Next, one example of this invention is explained using a drawing. It is the sectional view of a capillary tube [in / drawing 1 , and / in drawing 2 / drawing 1]. [the block diagram of the electrophoresis apparatus of one example of this invention]

0021] In drawing 1 , the 1st buffer-solution tub in which, as for 11, the migration buffer solution was stored, and 12 are the 2nd buffer-solution tubs in which the migration buffer solution was stored. The dipping of the platinum electrode 14 by the side of the cathode of the high-voltage generator 13 is carried out to the 1st buffer-solution tub 11, and the dipping of the platinum electrode 15 by the side of an anode plate is carried out to the 2nd buffer-solution tub 12.

0022] 16 is a capillary tube, a leader is made into the 1st buffer-solution tub 11, and the dipping of the trailer is carried out to the 2nd buffer-solution tub 12, respectively. The capillary tube 16 of this example consists of fuze DOSHIRIKA 17 and a polyimide enveloping layer 18 formed in the skin of fuze DOSHIRIKA 17.

0023] An enveloping layer 18 exfoliates and the detecting element 20 is formed by the 2nd buffer-solution tub 12 side of

capillary tube 16. UV detector 21 which consists of a UV and a diode array detector is formed in this detecting element 20.

0024] The migration buffer solutions, such as 2,6-pyridine dicarboxylic acid and nitrilotriacetic acid, are stored by the 1st and 2nd buffer-solution tubs 11 and 12. Drawing 3 is drawing showing the complex stability constant to the metal of the compound used for an organic acid or the migration buffer solution. For example, it turns out that 2,6-pyridine dicarboxylic acid has high complexing to the iron contained in fuze DOSHIRIKA 17, aluminum, sodium, and magnesium.

0025] Therefore, the 2,6-pyridine dicarboxylic acid which is the migration buffer solution when analyzing the sample of the anion nature compound which has high complex-forming properties, such as an organic acid (for example, oxalic acid, a citric acid, a malic acid, and ethylenediaminetetraacetic acid) and an inorganic anion, The metal (iron, aluminum, sodium, magnesium) contained in the capillary tube 16 forms a complex, the metal contained in the capillary tube 16 is masked, a metal and a complex are not formed, but even if a sample is low concentration, the high analysis of precision of the sample is attained.

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EXAMPLE

Example] The detail specification of the equipment of a configuration as shown in drawing 1 is described below.

1) Capillary tube 16 bore of 75 micrometers, the outer diameter of 350 micrometers, the overall length of 80.5cm, 72cm die length from the 1st buffer-solution tub 11 to a detecting element 20) (2) of effective length Buffer-solution 5mM 2,6-pyridine dicarboxylic acid, 0.5mM(s) Cetyl trimethylammonium star's picture pH 5.6 (3) Applied voltage of -25kV (4) (5) which pours the preconditioning migration buffer solution for 4 minutes Temperature of 20 degrees C (6) The impregnation approach inflatable flexible bag technique of a sample is used. 50mbars -- impregnation during 2 seconds 7) the UV detector 21 measurement wavelength of 200nm, and 350nm of reference wave length -- an indirect extinction method -- detection [experiment 1] -- the result of having analyzed the organic-acid standard solution which has the following organic-acid anions is shown in drawing 4.

0027] 1. Chloride Ion (Chloride) 2. Sulfate ion (sulfate) 3. Oxalate ion (oxalate) 4. Formic-acid ion (formate) 5. Malic-acid ion (malate) 6. Citric-acid ion (citrate) 7. Succinic-acid ion (succinate) 8. Pyruvic-acid ion (pyruvate) 9. Acetic-acid ion (acetate) 10. Lactic-acid ion (lactate) 11. Phosphoric-acid ion (phosphate) 12. As shown in pyroglutamic acid (pyroglutamate) drawing 5 When the 2,6-pyridine-dicarboxylic-acid buffer solution was used, the repeatability (relative standard deviation) of transit time was less than 0.13%, and the repeatability (relative standard deviation) of area (peak area) was also very good as 0.6 - 2.6%.

The organic acid in Biel diluted 5 times with water analyzes using 2,6-pyridine dicarboxylic acid as the [experiment 2] buffer solution, and a result is shown in drawing 6. Analysis of iron, aluminum, etc. the oxalate ion that is easy to form a complex, citric-acid ion, etc. was attained.

Experiment 3] The inorganic anion liquid of the following presentations is analyzed and a result is shown in drawing 7.

0028] 1. Chloride Ion (Cl-) 2. Sulfate Ion (SO42-) 3. Chloric-Acid Ion (ClO3-) 4. Bromate (BrO3-) 5. Chlorous-Acid Ion (ClO2-) 6. Hypophosphorous Acid Ion (PO23-) 7. Iodic-Acid Ion (IO3-) 8. phosphorous acid ion (PO33-) 9. phosphoric-acid ion (PO43-) -- by this approach, it became possible to measure these inorganic anions.

As the [experiment 4] migration buffer solution, using 2,6-pyridine dicarboxylic acid (PDC), and the phthalic acid and trimellitic acid which are used conventionally well to an organic acid, the organic-acid standard solution which used these comparisons in the experiment 1 performs, and a result is shown in drawing 8 (a) - (b).

0029] If 2,6-pyridine dicarboxylic acid is used for the migration buffer solution, as the experiment 1 explained, all organic-acid ion will be detected. On the other hand, when a phthalic acid and trimellitic acid were used for the migration buffer solution, by 50 or less mg/l, neither the oxalate ion of peak No.3 nor the citric-acid ion of No.6 was detected for the concentration of an organic acid. Furthermore, the peak of the malic-acid ion of No.5 also became small, and the effectiveness of 2,6-pyridine dicarboxylic acid was checked.

Translation done.]

NOTICES *

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3. In the drawings, any words are not translated.

DESCRIPTION OF DRAWINGS

Brief Description of the Drawings]

Drawing 1] It is the block diagram of the electrophoresis apparatus of one example of this invention.

Drawing 2] It is the sectional view of the capillary tube in drawing 1 .

Drawing 3] It is drawing showing the complex stability constant to the metal of the compound used for an organic acid or the migration buffer solution.

Drawing 4] It is drawing showing the result of experiment 1.

Drawing 5] It is drawing showing the result of experiment 1.

Drawing 6] It is drawing showing the result of experiment 2.

Drawing 7] It is drawing showing the result of experiment 3.

Drawing 8] It is drawing showing the result of experiment 4.

Drawing 9] It is the block diagram of conventional capillary-electrophoresis equipment.

Drawing 10] It is drawing explaining the trouble of conventional capillary-electrophoresis equipment.

Drawing 11] It is drawing showing the metal ion concentration in a fuze DOSHIRIKA capillary tube.

Description of Notations]

11 1st Buffer-Solution Tub

12 2nd Buffer-Solution Tub

13 High-Voltage Generator

14 15 Electrode

16 Capillary Tube

20 Detecting Element

21 UV Detector

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NOTICES *

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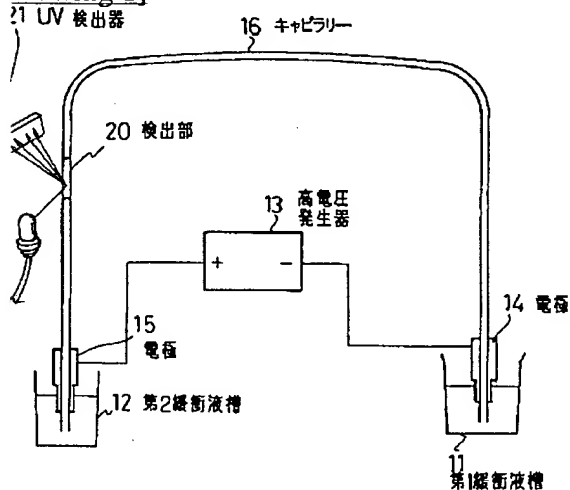
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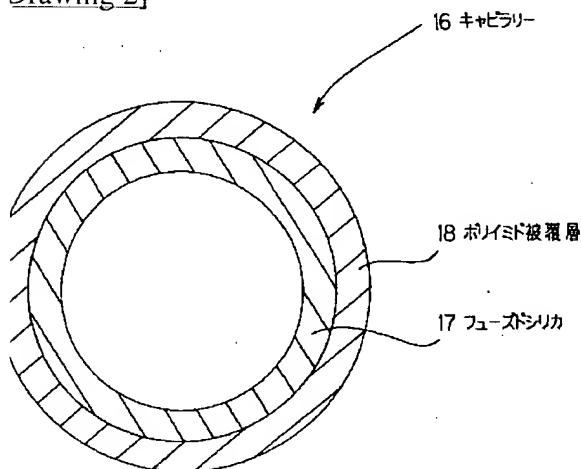
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DRAWINGS

Drawing 1]



Drawing 2]



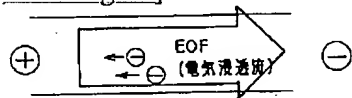
Drawing 3]

	錯安定度定数 LogK1					
	Pb ²⁺	Fe ³⁺	Al ³⁺	Na ⁺	Cr ²⁺	Mg ²⁺
シュウ酸 (Oxalate)	8.06	7.53	5.97		3.85	2.76
クエン酸 (Citrate)	4.80	11.50	7.98	0.71		3.45
リンゴ酸 (Malate)	2.60	7.10		0.29		1.71
ギ酸 (Formate)		3.10	1.38		1.07	0.94
コハク酸 (Succinate)	1.40	6.88	3.20	0.08		1.18
ピルビン酸 (Pyruvate)				0.20		1.10
酢酸 (Acetate)	1.40	3.38	1.51	-0.26	1.25	0.55
乳酸 (Lactate)				1.66		0.93
2,6-ピジジン (PDC)	5.71	10.91	4.87			2.91
カルボン酸						
フタル酸 (Phthalate)			3.18			
安息香酸 (Benzoate)			12.09			0.10
ピロバット酸 (Pyromellitate)						
トリバット酸 (Trimellitate)						
エチレンジアミン (EDTA)	14.30	25.10		1.84	13.60	8.85
四酢酸						

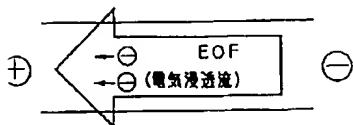
Drawing 5]

陰イオン	相対標準偏差(n=6)(%)		検量線の直線性	検出下限 (mg l ⁻¹)
	移動時間	ピークエリア		
Chloride (塩化物イオン)	0.10	1.8	0.9997	1.9
Sulfate (硫酸イオン)	0.12	2.6	0.9999	1.4
Oxalate (シュウ酸イオン)	0.11	1.5	0.9993	1.8
Formate (ギ酸イオン)	0.11	1.7	0.9994	1.0
Malate (リンゴ酸イオン)	0.12	1.8	0.9993	1.2
Citrate (クエン酸イオン)	0.12	2.2	0.9997	2.2
Succinate (コハク酸イオン)	0.13	1.1	0.9997	1.2
Pyruvate (ピルビン酸イオン)	0.12	2.5	0.9997	2.4
Acetate (酢酸イオン)	0.12	1.5	0.9996	0.9
Lactate (乳酸イオン)	0.13	0.6	0.9992	1.2
Phosphate (リン酸イオン)	0.13	2.0	0.9999	1.8
Pyroglutamate (ピコグルタミン酸イオン)	0.13	1.7	0.9995	2.5

Drawing 10]

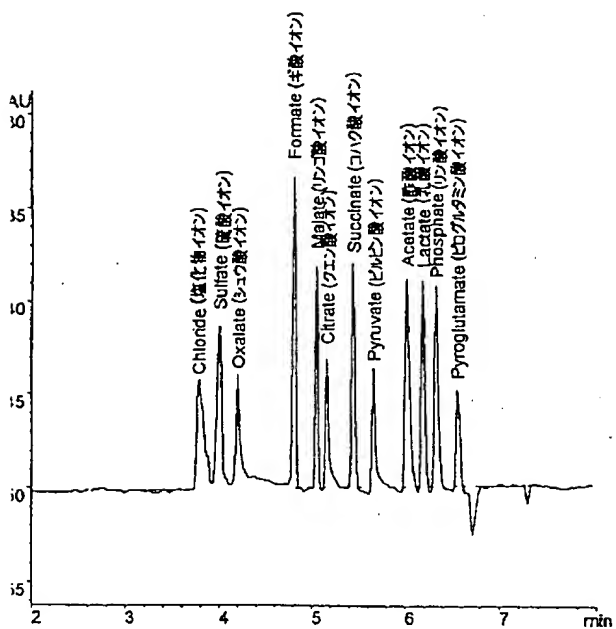


(a)

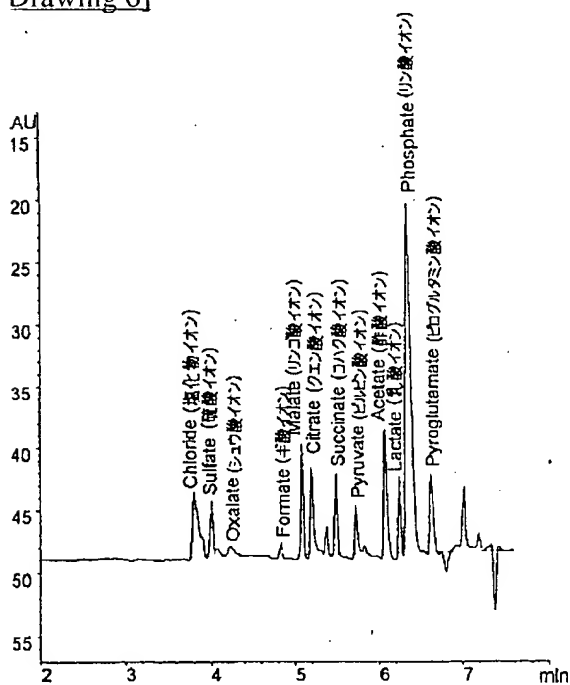


(b)

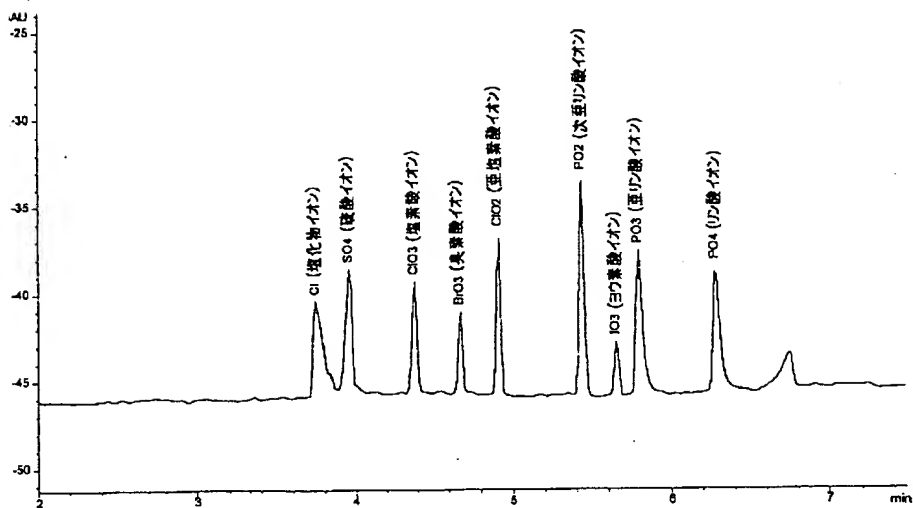
[Drawing 4]



Drawing 6]



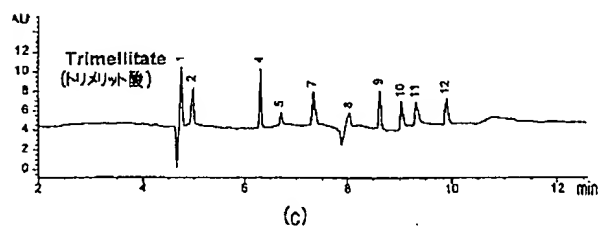
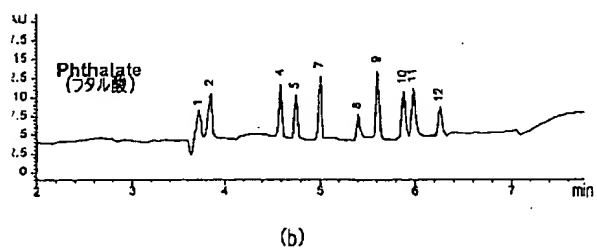
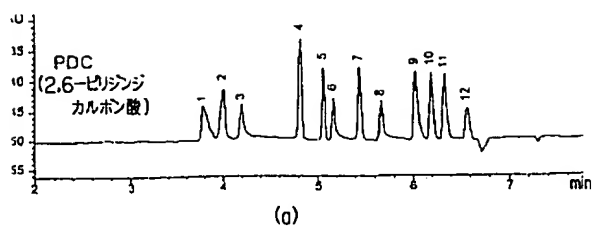
Drawing 7]



Drawing 11]

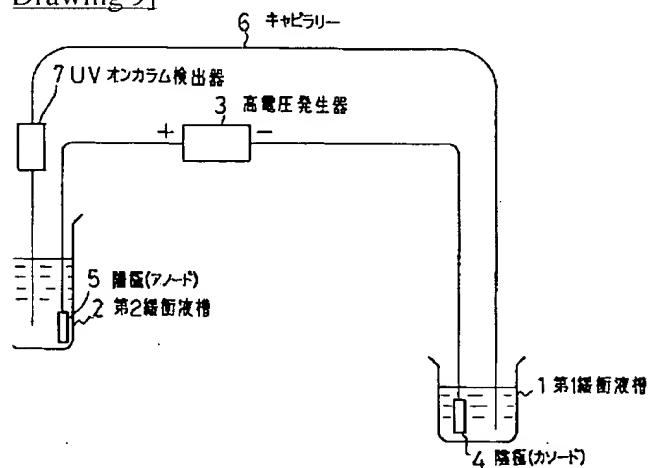
金 属	濃 度 (ppb)
Fe (鉄)	300
Al (アルミニウム)	240
Na (ナトリウム)	140
Cr (クロム)	40
Mg (マグネシウム)	30

Drawing 8]



1. 塩化物イオン(chloride)
2. 硫酸イオン(sulfate)
3. シュウ酸イオン(oxalate)
4. フォルミオン(formate)
5. リンゴ酸イオン(malate)
6. クエン酸イオン(citrate)
7. コハク酸イオン(succinate)
8. ピルビン酸イオン(pyruvate)
9. 酢酸イオン(acetate)
10. 乳酸イオン(lactate)
11. リン酸イオン(phosphate)
12. ピログルタミン酸イオン(pyroglutamate)

Drawing 9]



[Translation done.]

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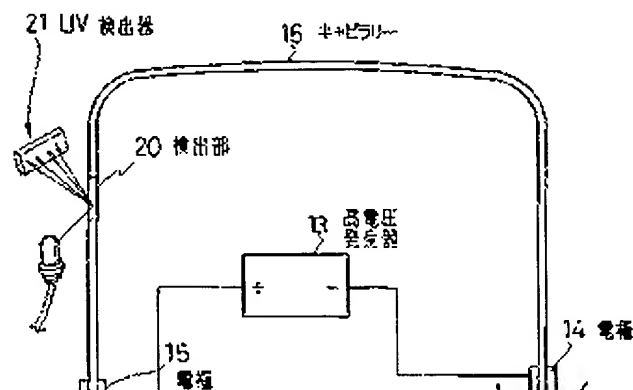
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(54) 【発明の名称】 陰イオン性化合物の分析方法

(57) 【要約】

【課題】 金属と錯形成能が高い低濃度陰イオン性化合物の分析方法及び分析装置を提供することを課題とする。

【解決手段】 キャピラリー16に泳動緩衝液を満たし、キャピラリー16の両端に高電圧を印加し、キャピラリー16の一方の端部から導入される金属に対して高い錯形成能を有する陰イオン性化合物の分析を行なう場合、泳動緩衝液をキャピラリー16に含まれる金属に対して高い錯形成能を有する陰イオン性物質とする。



【特許請求の範囲】

【請求項1】 キャピラリーに泳動緩衝液を満ちし、前記キャピラリーの両端に高電圧を印加し、前記キャピラリーの一方の端部から導入される金属に対して高い錯形成能を有する陰イオン性化合物の分析を行なう方法において、

前記泳動緩衝液を前記キャピラリーに含まれる金属に対して高い錯形成能を有する陰イオン性物質とすることを特徴とする陰イオン性化合物の分析方法。

【請求項2】 前記キャピラリーの材質がフューズドシリカで、前記泳動緩衝液が2,6-ピリジンジカルボン酸であることを特徴とする請求項1記載の陰イオン性化合物の分析方法。

【請求項3】 前記キャピラリーの材質がフューズドシリカで、前記泳動緩衝液がニトリロ三酢酸であることを特徴とする請求項1記載の陰イオン性化合物の分析方法。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】 本発明は、キャピラリーに泳動緩衝液を満ちし、前記キャピラリーの両端に高電圧を印加し、前記キャピラリーの一方の端部から導入された有機酸及び無機イオン等の金属に対して高い錯形成能を有する陰イオン性化合物の分析を行なう分析方法に関する。

【0002】

【従来の技術】 周知の如く、キャピラリーを用いたキャピラリー電気泳動法は、ナノリットル(nl)乃至ピコリットル(pl)もの極微量の試料を測定対象とし、現在、最も高い分離能力をもつ分析手段として注目されている。

【0003】 図9は従来のキャピラリー電気泳動装置の構成図である。図において、1は泳動緩衝液が貯留された第1緩衝液槽、2は泳動緩衝液が収容された第2緩衝液槽である。

【0004】 高電圧発生器3の陰極(カソード)4は第1緩衝液槽1に浸せきされ、陽極(アノード)5は第2緩衝液槽2に浸せきされている。6は例えば、フューズドシリカ等のキャピラリーで、始端部は第1緩衝液槽1に、終端部は第2緩衝液槽2にそれぞれ浸せきされている。

【0005】 キャピラリー6の第2緩衝液槽2側には、

動する試料成分は、水和した試料のイオン半径やイオン性の違いに応じた移動時間を有し、これをイオンカラム検出器7で検出する。

【0007】

【発明が解決しようとする課題】 上記構成のキャピラリー電気泳動装置において、試料が有機酸や無機陰イオン等のように強いU吸収性を持たない陰イオン性化合物の場合は図10(a)に示すように、電気浸透流(以下、ECFという)と反対に泳動するので、分析時間がかかるか、検出することができなかった。

【0008】 そこで、特開平6-94677号公報では、フタル酸、トリメリット酸やクロム酸等の泳動緩衝液にセチルトリメチルアンモニウムブロマイド等の4級アミン類を添加し、図10(b)に示すようにECFを反転させる方法が開示されている。

【0009】 このような方法においては、泳動緩衝液のU吸収のためバックグラウンドは、一定となっているが、強いU吸収を持たない被検出物が通過すると、吸光度が低くなり、負のピークとなる。この負のピークの信号を反転させ、正のピークにして検出する。

【0010】 とところで、一般に、フューズドシリカのキャピラリー6には、不純物が含まれている。図11は、誘導結合プラズマ質量分析装置(ICP-MS)で測定したフューズドシリカキャピラリー6中の金属イオン濃度を示す図である。この図より、フューズドシリカキャピラリー6には、鉄(Fe)、Al(アルミニウム)、Na(ナトリウム)、Cr(クロム)、Mg(マグネシウム)等の金属が不純物として含まれていることが分かる。

【0011】 ここで、泳動緩衝液として、上記したフタル酸、トリメリット酸やクロム酸等を用い、試料が有機酸(例えば、シュウ酸、クエン酸、リンゴ酸やエチレンジアミン四酢酸)や無機陰イオン等の高い錯形成能を有する陰イオン性化合物の場合、試料濃度が約50ppm以下の低濃度になると、キャピラリー6のフューズドシリカに不純物として含まれている鉄やアルミニウム等の金属と、これら陰イオン性化合物とが錯体を形成し、試料の分析ができないという問題点がある。

【0012】 本発明は上記問題点に鑑みてなされたもので、その目的は、金属と錯形成能が強い低濃度陰イオン性化合物の分析方法及び分析装置を提供することにあ

能を有する陰イオン性物質とする陰イオン性化合物の分析方法である。

【0014】泳動緩衝液が、前記キャピラリーに含まれる金属に対して高い錯形成能を有する陰イオン性物質なので、キャピラリーに含まれている金属と強く錯体を形成し、これらの金属をマスキングしてしまう。

【0015】よって、試料が金属と高い錯形成能を有していても、試料は金属と錯体を形成せず、試料が低濃度であっても精度の高い分析が可能となる。

(2) 第2の発明

第1の発明において、前記キャピラリーの材質がフューズドシリカで、前記泳動緩衝液が2,6-ピリジンジカルボン酸である陰イオン性化合物の分析方法である。

【0016】キャピラリーに用いられるフューズドシリカに含まれている金属としては、鉄、アルミニウム等がある。一方、2,6-ピリジンジカルボン酸は、これら鉄、アルミニウム等に対して高い錯安定度数を有しており、泳動緩衝液に用いることでこれらの金属をマスキングしてしまう。

【0017】よって、試料が金属と高い錯形成能を有していても、試料は金属と錯体を形成せず、試料が低濃度であっても精度の高い分析が可能となる。

(3) 第3の発明

第1の発明において、前記キャピラリーの材質がフューズドシリカで、前記泳動緩衝液がニトリロ三酢酸である陰イオン性化合物の分析方法である。

【0018】フューズドシリカキャピラリーに含まれている金属としては、鉄、アルミニウム等がある。一方、ニトリロ三酢酸は、これら鉄、アルミニウム等に対して高い錯安定度数を有しており、泳動緩衝液に用いることでこれらの金属をマスキングしてしまう。

【0019】よって、試料が金属と高い錯形成能を有していても、試料は金属と錯体を形成せず、試料が低濃度であっても精度の高い分析が可能となる。

【0020】

【発明の実施の形態】次に、図面を用いて本発明の一実施例を説明する。図1は本発明の一実施例の電気泳動装置の構成図、図2は図1におけるキャピラリーの断面図である。

【0021】図1において、11は泳動緩衝液が貯留さ

は、被覆層18が剥離され、検出部20が形成されている。この検出部20には、UVおよびダイオードアレイ検出器からなるUV検出器21が設けられている。

【0024】第1及び第2緩衝液槽11、12には、2,6-ピリジンジカルボン酸やニトリロ三酢酸等の泳動緩衝液が貯留されている。図3は、有機酸や泳動緩衝液に使用する化合物の金属に対する錯安定度定数を示す図である。例えば、2,6-ピリジンジカルボン酸は、フューズドシリカ17に含まれている鉄、アルミニウム、ナトリウム、マグネシウムに対して高い錯形成を有していることが分かる。

【0025】よって、有機酸(例えば、シュウ酸、クエン酸、リンゴ酸やエチレンジアミン四酢酸)や無機陰イオン等の高い錯形成能を有する陰イオン性化合物の試料を分析する場合、泳動緩衝液である2,6-ピリジンジカルボン酸と、キャピラリー16に含まれている金属(鉄、アルミニウム、ナトリウム、マグネシウム)とが錯体を形成し、キャピラリー16に含まれている金属はマスキングされ、試料は金属と錯体を形成せず、試料が低濃度であっても精度の高い分析が可能となる。

【0026】

【実施例】図1に示すような構成の装置の詳細仕様を以下に記す。

(1) キャピラリー16

内径75 μ m、外径350 μ m、全長80.5cm、有効長(第1の緩衝液槽11から検出部20までの長さ)72cm

(2) 緩衝液

5mM 2,6-ピリジンジカルボン酸、0.5mM セチルトリメチルアンモニウムブロマイド

pH5.6

(3) 印加電圧

-25kV

(4) プレコンディショニング

泳動緩衝液を4分間流す

(5) 温度

20℃

(6) 試料の注入方法

加圧法を用いて、50mbarで2秒間注入

(7) UV検出器21

測定波長200nm、発光波長350nmで間接吸光度法で検出

9. 酢酸イオン(acetate)
10. 乳酸イオン(lacate)
11. リン酸イオン(phosphate)
12. ピログルタミン酸(pyroglutamate)

図5に示すように、2,6-ピリジンジカルボン酸緩衝液を用いると、移動時間の再現性(相対標準偏差)が0.1%以内であり、また、面積(ピークエリア)の再現性(相対標準偏差)も0.6~2.6%と非常に良好であった。

【実験2】緩衝液として2,6-ピリジンジカルボン酸を用い、水で5倍に希釈したビール中の有機酸の分析し、結果を図6に示す。鉄やアルミニウム等と錯体を形成しやすいシュウ酸イオンやクエン酸イオン等が分析可能となった。

【実験3】以下のような組成の無機陰イオン液を分析し、結果を図7に示す。

- 【0028】1. 塩化物イオン(Cl^-)
2. 硫酸イオン(SO_4^{2-})
 3. 塩素酸イオン(ClO_3^-)
 4. 臭素酸イオン(BrO_3^-)
 5. 亜塩素酸イオン(ClO_2^-)
 6. 次亜リン酸イオン(PO_2^{3-})
 7. ヨウ素酸イオン(IO_3^-)
 8. 亜リン酸イオン(PO_2^{3-})
 9. リン酸イオン(PO_4^{3-})

この方法では、これら無機陰イオン類も測定することが可能となった。

【実験4】泳動緩衝液として、2,6-ピリジンジカルボン酸(PDC)と、従来有機酸によく用いられるフタル酸、トリメリット酸とを用い、これらの比較を実験1で用いた有機酸標準液で行い、結果を図8(a)~(b)に示す。

【0029】2,6-ピリジンジカルボン酸を泳動緩衝液に用いると、実験1で説明を行ったように、すべての有機酸イオンが検出される。一方、フタル酸、トリメリット酸を泳動緩衝液に用いると、有機酸の濃度が50mg/l以下では、ピークNo.3のシュウ酸イオンや、No.6のクエン酸イオンが検出されなかった。更に、No.5のリンゴ酸イオンのピークも小さくなり、2,6-ピリジンジカルボン酸の有効性が確認された。

【0030】

【発明の効果】以上詳しく説明したように、第1の発明

であっても精度の高い分析が可能となる。第2の発明の陰イオン性化合物の分析方法によれば、第1の発明において、前記キャピラリーの材質がフューズドシリカで、前記泳動緩衝液が2,6-ピリジンジカルボン酸とした。キャピラリーに用いられるフューズドシリカに含まれている金属としては、鉄、アルミニウム等がある。一方、2,6-ピリジンジカルボン酸は、これら鉄、アルミニウム等に対して高い錯安定度数を有しており、これらの金属をマスキングしてしまう。

【0032】よって、試料が金属と高い錯形成能を有していても、試料は金属と錯体を形成せず、試料が低濃度であっても精度の高い分析が可能となる。第3の発明の陰イオン性化合物の分析方法によれば、第1の発明において、前記キャピラリーの材質がフューズドシリカで、前記泳動緩衝液がニトリロ三酢酸とした。

【0033】フューズドシリカキャピラリーに含まれている金属としては、鉄、アルミニウム等がある。一方、ニトリロ三酢酸は、これら鉄、アルミニウム等に対して高い錯安定度数を有しており、マスキングしてしまう。

【0034】よって、試料が金属と高い錯形成能を有していても、試料は金属と錯体を形成せず、試料が低濃度であっても精度の高い分析が可能となる。

【図面の簡単な説明】

【図1】本発明の一実施例の電気泳動装置の構成図である。

【図2】図1におけるキャピラリーの断面図である。

【図3】有機酸や泳動緩衝液に使用する化合物の金属に対する錯安定度定数を示す図である。

【図4】実験1の結果を示す図である。

【図5】実験1の結果を示す図である。

【図6】実験2の結果を示す図である。

【図7】実験3の結果を示す図である。

【図8】実験4の結果を示す図である。

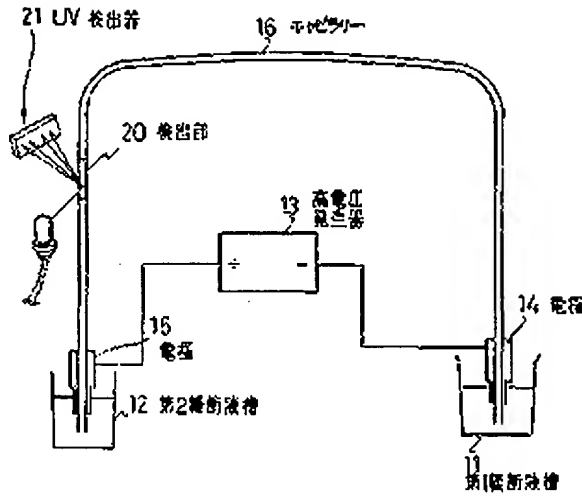
【図9】従来のキャピラリー電気泳動装置の構成図である。

【図10】従来のキャピラリー電気泳動装置の問題点を説明する図である。

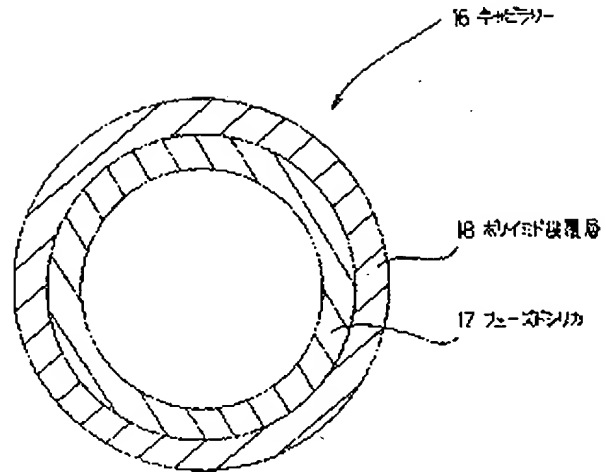
【図11】フューズドシリカキャピラリー中の金属イオン濃度を示す図である。

【符号の説明】

【図1】



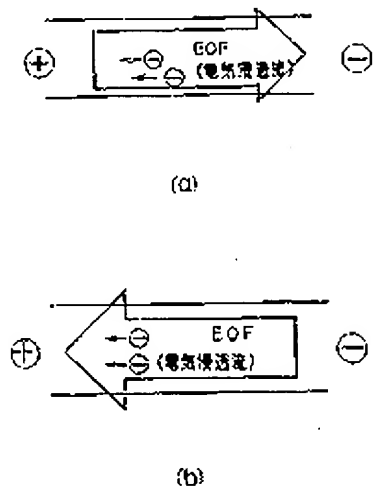
【図2】



【図3】

	安定定数 $\log K_1$					
	Co^{2+}	Fe^{3+}	Al^{3+}	Na^{+}	Cl^{-}	Br^{-}
シュウ酸 (Oxalate)	3.03	7.58	5.47		0.85	2.76
クエン酸 (Citrate)	4.80	11.50	7.98	0.71		3.45
リンゴ酸 (Malate)	2.00	7.10		0.20		1.71
酢酸 (Formate)		3.10	1.38		1.07	0.84
コハク酸 (Succinate)	1.40	0.28	3.20	0.00		1.18
ピルビン酸 (Pyruvate)				0.20		1.10
酢酸 (Acetate)	1.40	0.28	1.61	-0.26	1.25	0.55
乳酸 (Lactate)				1.68		0.99
2,6-ピリジン カルボン酸 (PDC)	5.71	10.91	4.83			2.01
フタル酸 (Phthalate)			3.18			
安息香酸 (Benzoate)			12.09			0.10
ピロキソリ酸 (Pyrooxalate)						
トリメチル酸 (Trimellitate)						
エチレンジ 酸 (EDTA)	14.80	25.18		1.84	13.60	3.86

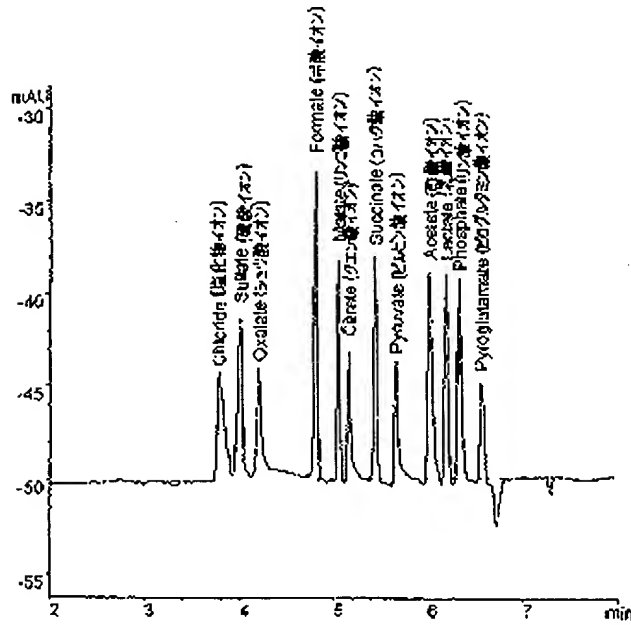
【図4】



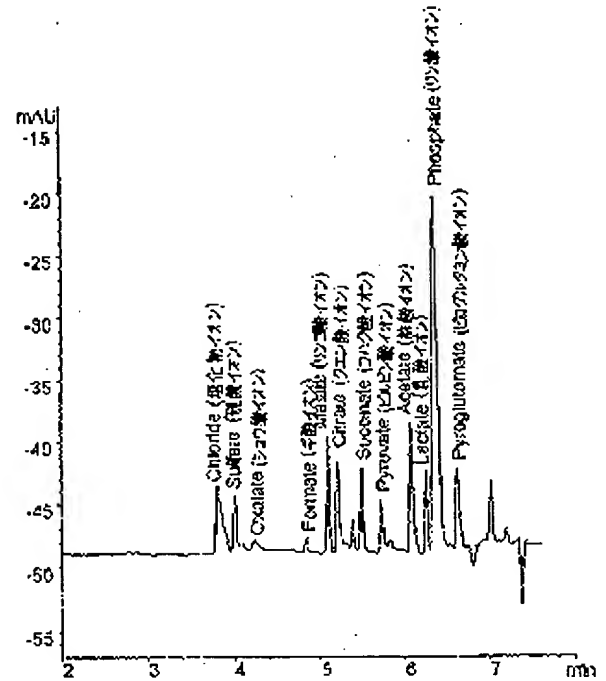
【図5】

陰イオン	相対標準偏差 (%)		検量線の 直線性	検出下限 (mg/L)
	移動時間	ピークエリア		
Chloride (塩化陰イオン)	0.10	1.8	0.9997	1.0

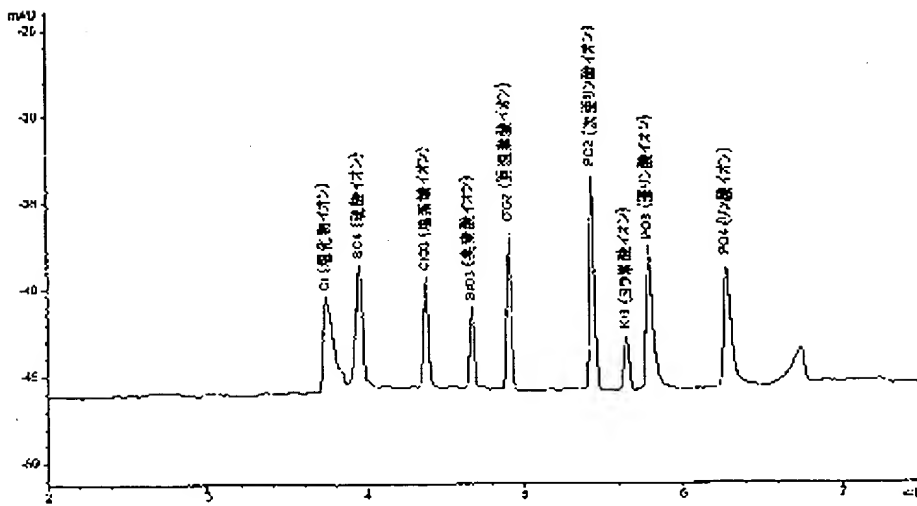
【図4】



【図6】



【図7】

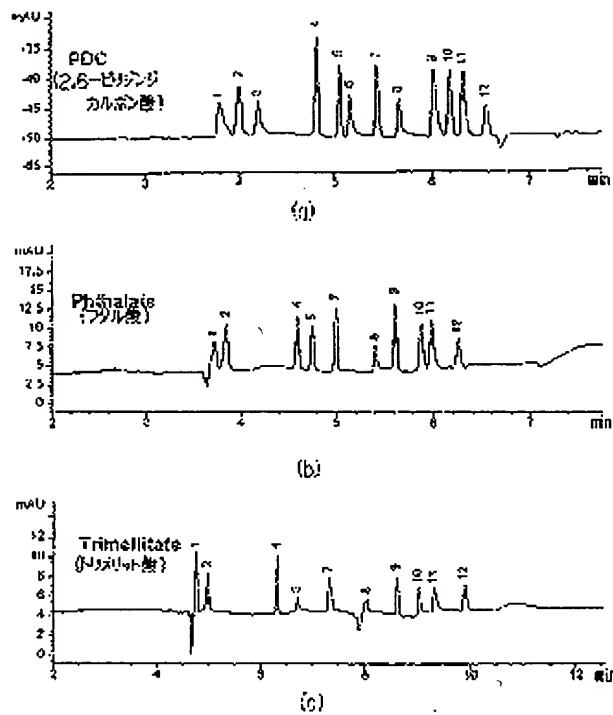


【図11】

(7)

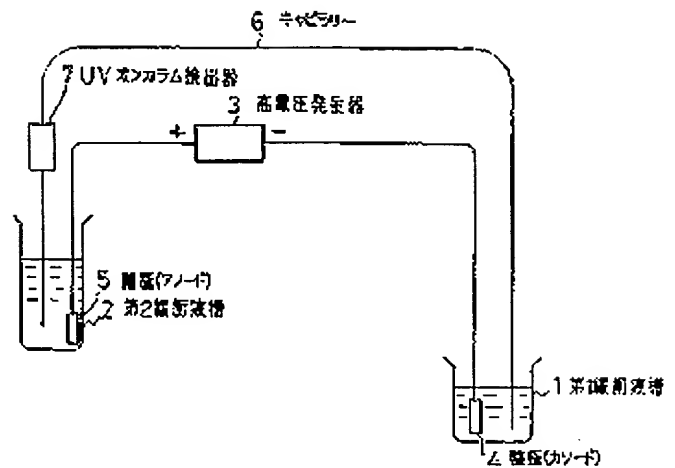
特開平9-325130

【図8】



1. 塩化物イオン(chloride)
2. 硫酸イオン(sulfate)
3. シュウ酸イオン(oxalate)
4. 酢酸イオン(formate)
5. リンゴ酸イオン(malate)
6. クエン酸イオン(citrate)
7. コハク酸イオン(succinate)
8. ピルビン酸イオン(pyruvate)
9. 酢酸イオン(acetate)
10. 乳酸イオン(lactate)
11. リン酸イオン(phosphate)
12. ピログルタミン酸イオン(pyroglutamate)

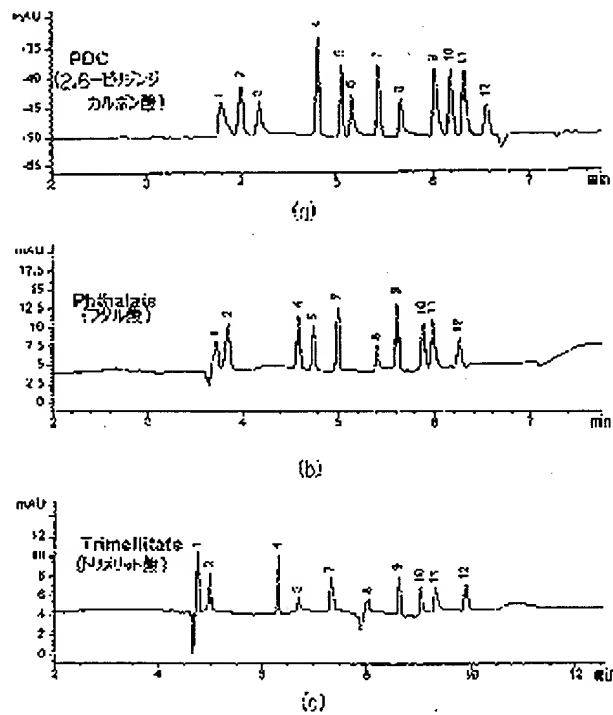
【図9】



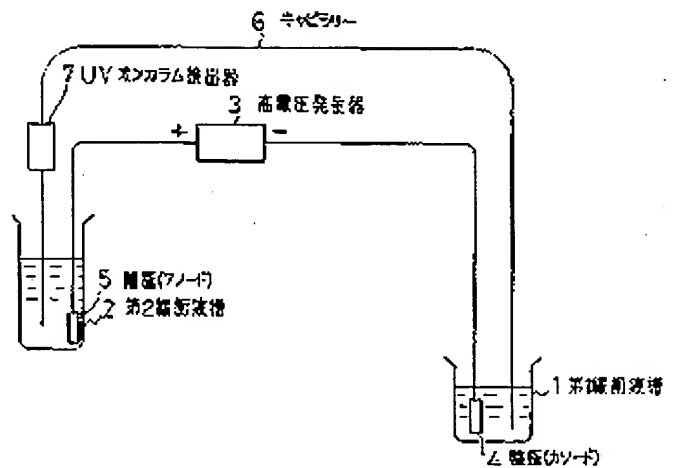
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【図8】



【図9】



1. 塩化物イオン(chloride)
2. 硫酸イオン(sulfate)
3. シュウ酸イオン(oxalate)
4. 酢酸イオン(formate)
5. リンゴ酸イオン(malate)
6. クエン酸イオン(citrate)
7. コハク酸イオン(succinate)
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9. 酢酸イオン(acetate)
10. 乳酸イオン(lactate)
11. リン酸イオン(phosphate)
12. ピログルタミン酸イオン(pyroglutamate)